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Recovery of Monomer from PET Waste Powder for Recycling

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ABSTRACT

Recovery of monomer terepthalic acid (TPA) from the polythelene terepthalic acid (PET) waste powder was carried out by degradation of PET waste powder of drinking bottles. The molecular weight of PET waste powder was found to be 8851. The minimum amount of sodium hydroxide and pyridine required for maximum recovery was found to be 7g. and 3.0cm^3 respectively. Further, it was observed that minimum particle size of 100 μm of PET waste powder gave maximum conversion to TPA. The maximum time and temperature required was 120 minute and 140^oC respectively.

Keywords: PET waste bottles, Terepthalic acid, degradation, recovery of monomer, hydrolysis.

INTRODUCTION

Presently disposal of polymer waste has been great environmental problem, because of difficulties arising in the set up and intermediate treatment plants as an incinerator or availability of land for reclamation. Polymer materials like PET bottles disposed by incineration and reclamation in many countries. Due to high transportation cost and their large volume has created serious social problems. Hence it is need of time to develop a new process for recycling of PET waste bottles.

PET bottles does not have any side effects on the metabolism of human being. Hence it has wide application in the manufacture of packaging for food industry.[1]

Recently environmentalist has focused on the recycling and recovery of monomers from packaging waste. PET containers are of visible size and part of domestic waste. Hence it is one if the prime material to be recovered and recycled.

Though PET recycling is commonly important there are numbers of difficulties encountered in the recycling of PET waste.[2-5]

In developed countries Government provides subsidies for collection and separation of PET waste; while in India it is done by individuals, dealers and manufacturers. Many researchers have reviewed the upgradation on plastic and environmental progress and trends.[6-9]

Now a days chemical recycling of PET waste has drawn greater attention because of valuable product obtained from this waste[10]. Various chemical agent used to depolymerise the PET waste are water, ethylene glycol, methanol butanediol, sodium hydroxide, sulphuric acid, nitric acid, phosphoric acid and ammonia.

Above reasons shows the considerable importance of recovery of monomer from PET waste powder.

EXPERIMENTAL

Material, Chemicals and Reagents

The PET mineral water bottles were procured and crushed into a fine powder of various particle size, ranging from 100 μm to 800 μm. Other materials used were sodium hydroxide, tetra chloroethane, phenol, pyridine supplied by S. D. fine chemicals (India). All the chemicals used were of analytical grade and used as received. Doubly glass distilled water was used throughout all the experiments.

Determination of molecular weight

The viscocity average molecular weight of PET waste powder was determined using Ostwald viscometer. The flow time of various solution prepared in solvent phenol and tetrachloroethene 3:5(v/v) was recorded. The intrinsic viscocity of the graph of $\eta_{sp/c}$ against the concentration was used to calculate molecular weight of PET waste powder using the formula.

$$[\eta_{sp/c}]=KM^{\alpha}$$

The values of constant K and α are 23.0×10^{-3} and 0.73 respectively (Ref. Fig. 1)

Conversion of PET waste powder into TPA

Conversion of PET waste powder was carried out in 250 cm^3 of round bottom flask equipped with reflux water condesor heating assembly at 1 atmospheric pressure and temperature 80^oc-160^oc. 10 gm of PET waste powder of different sizes, 2-9 g. of sodium hydroxide, 1.0-5.0 cm^3 of pyridine and 100cm^3 of double distilled water was charged in the round bottom flask. Them the mixture was reflux for the various time intervals such as 30-150 minutes. The reaction mixture was filtered for the separation of unreached PET waster powder. The filtrate was treated with concentrated hydrochloric acid till the solution becomes acidic in nature. A white precipitate of terepthalic acid (TPA) was obtained. It was washed repeatadely with double distilled water till it is completely free from hydrochloric acid. The complete removal of hydrochloric acid was tested by adding dilute silver nitrated solution. The white precipitated of terepthalic acid was dried and weighted. The terepthatic acid obtained was crushed into a fine powder and characterised

by taking FTIR spectra. It is also characterized by taking its melting point. Finally the FTIR spectra of authentic terepthalic acid was also taken to verify the terepthalic acid obtained by conversion of PET waste powder.

Table 1: Conversion of PET waste powder to terepthalic acid; variation of amount of sodium hydroxide.

Sr. No.	Amount of NaoH/gm	Percentage Conversion to TPA
1.	2.0	26.0
2.	3.0	39.0
3.	4.0	52.0
4.	5.0	65.0
5.	6.0	77.5
6.	7.0	87.0
7.	8.0	85.0
8.	9.0	75.0

Table 2: Conversion of PET waste powder to terephatic acid; variation of particle size

Sr. No.	Particle size of PET Waste/um	Percentage Conversion to TPA
1.	100	90.0
2.	200	88.5
3.	300	86.0
4.	400	80.6
5.	500	69.0
6.	600	60.0
7.	700	55.0
8.	800	52.0

Table 3 : Conversion of PET waste powder to terepthatic acid; variation of Reflux time

Sr. No.	Reflux time/min	Percentage Conversion to TPA
1.	30	22.5
2.	60	50.0
3.	90	66.0
4.	120	73.0
5.	150	73.0
6.	180	72.5

Table 4 : Conversion of PET waste powder to terepthatic acid; variation of Temperature

Sr. No.	Temperature/^{o}C	Percentage Conversion to TPA
1.	60	8.0
2.	80	48.5
3.	100	71.0
4.	120	83.5
5.	140	88.5
6.	160	87.5
7.	170	85.0

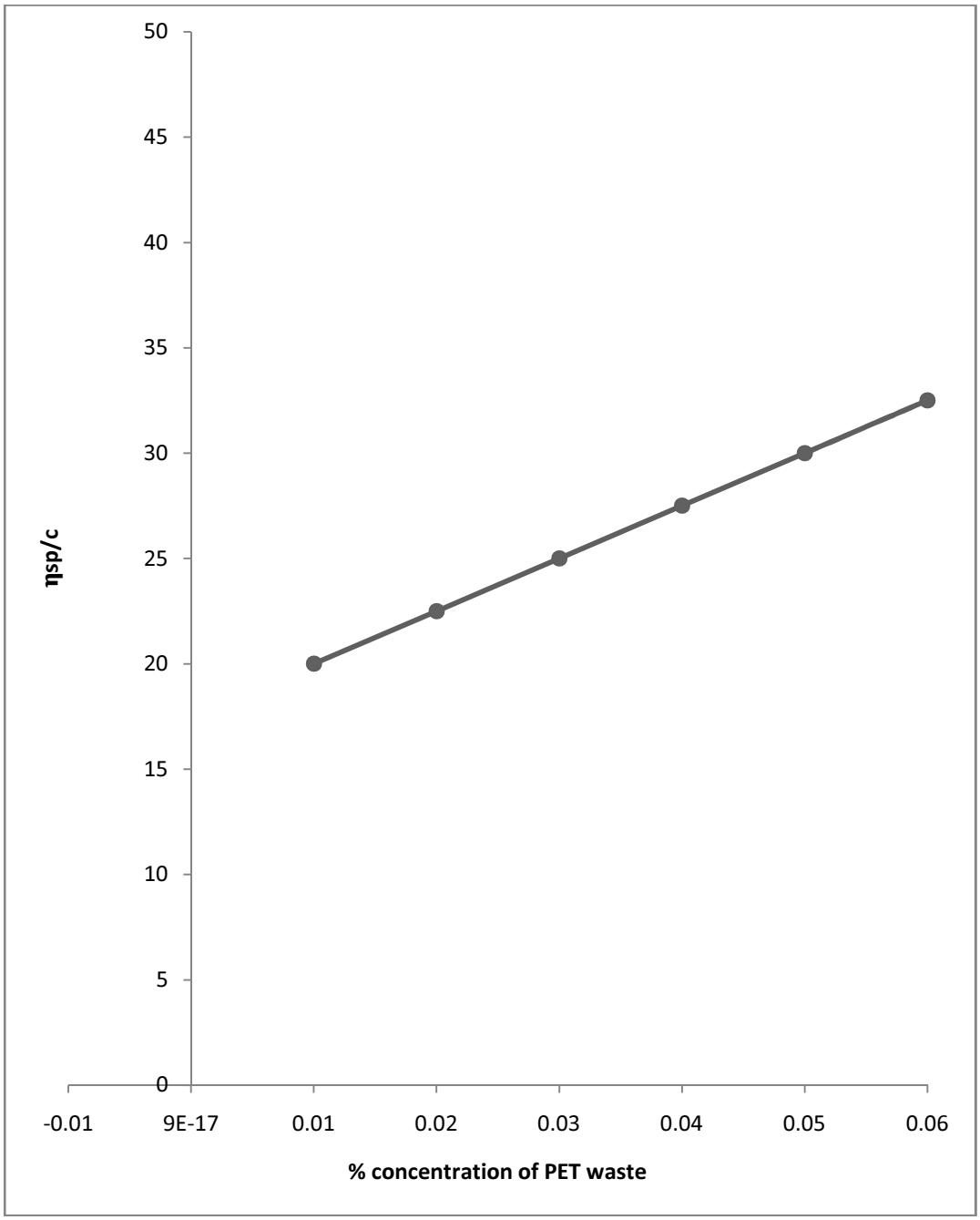


Fig. 1: Determination of Average molecular weight of PET waste

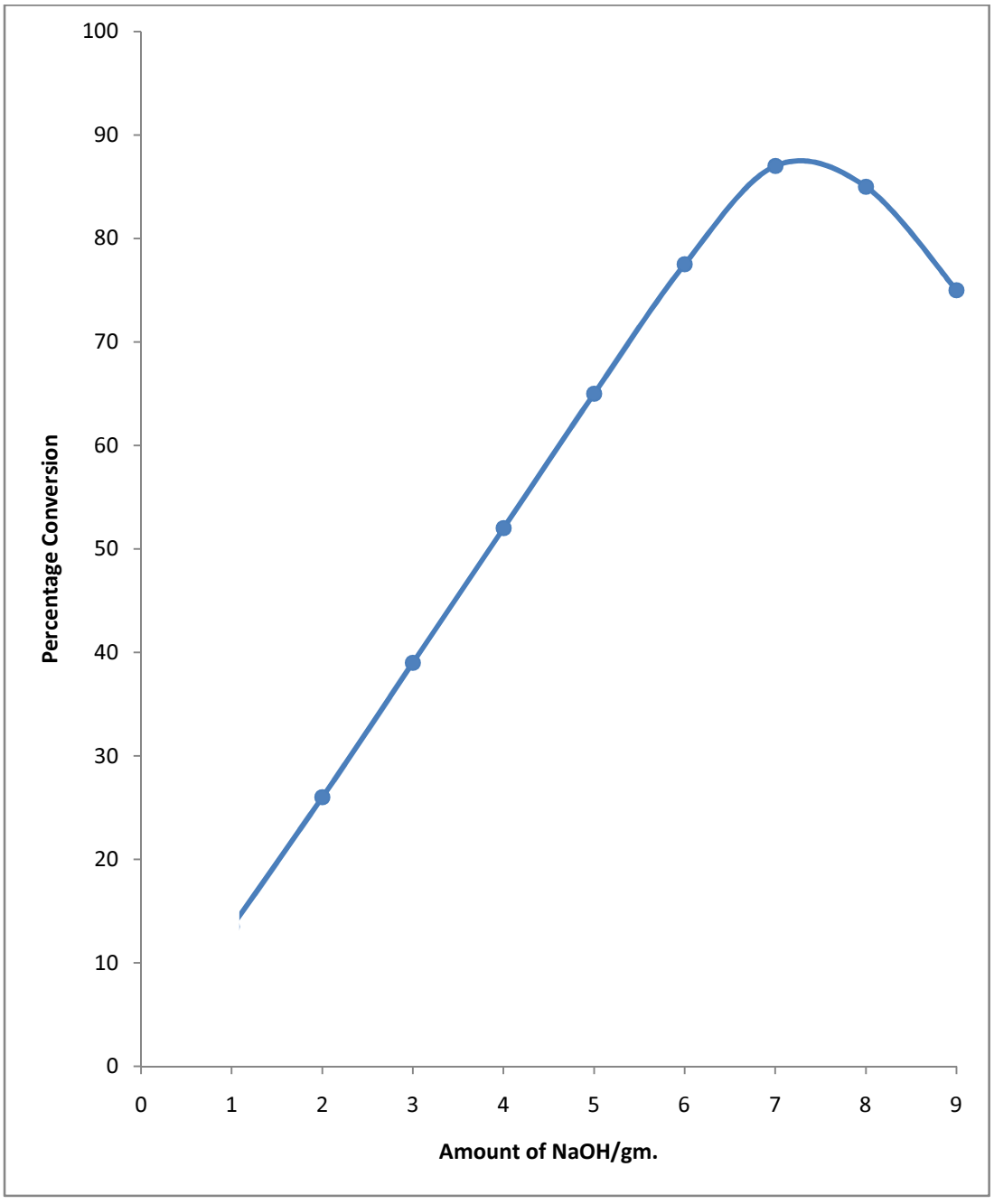


Fig. 2: Conversion of PET waste powder to terepthalic acid: variation of amount of sodium hydroxide

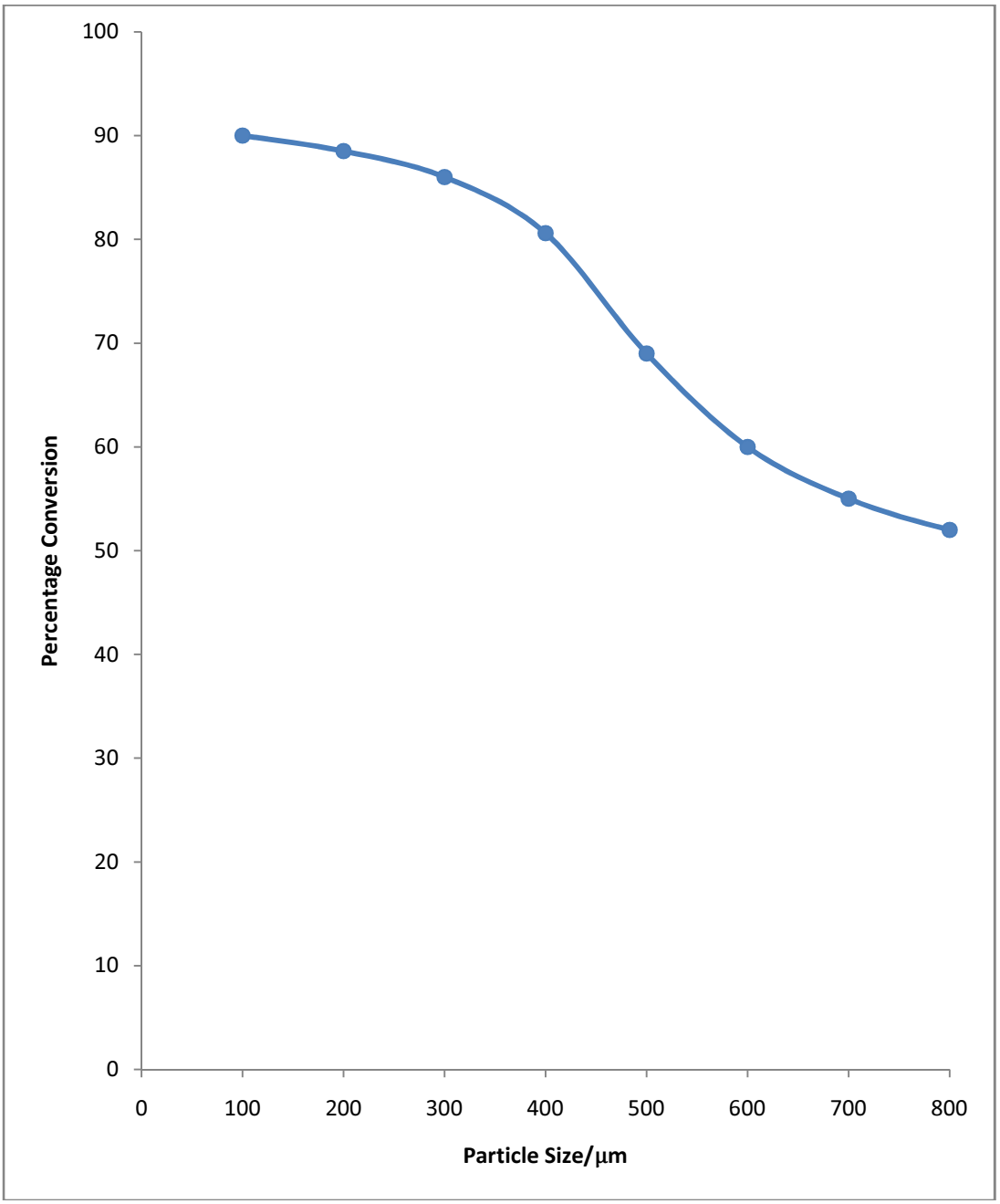


Fig. 3: Conversion of PET waste powder to terepthalic acid: variation of particle size

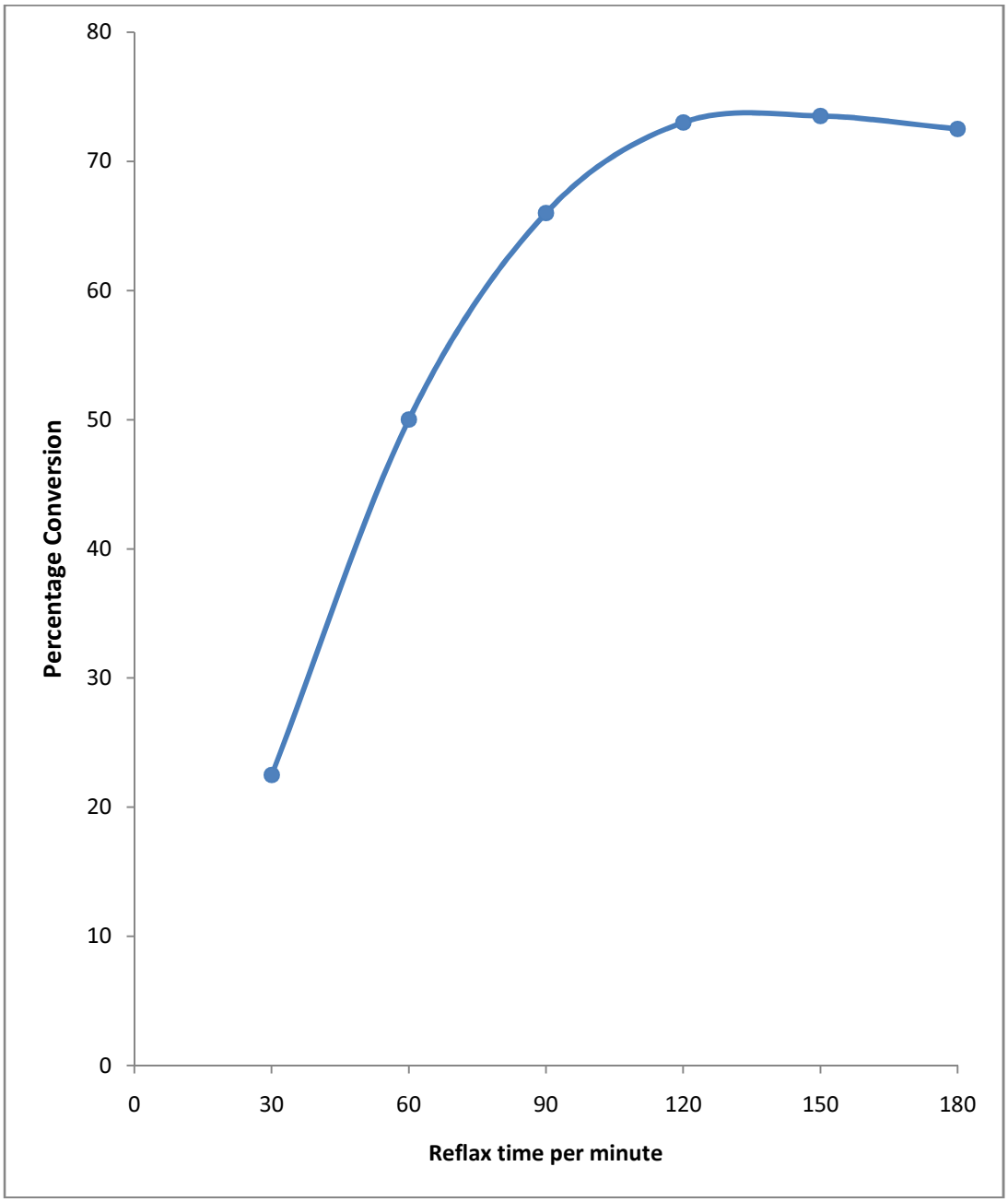


Fig. 4: Conversion of PET waste powder to terepthalic acid: Variation of reflex time

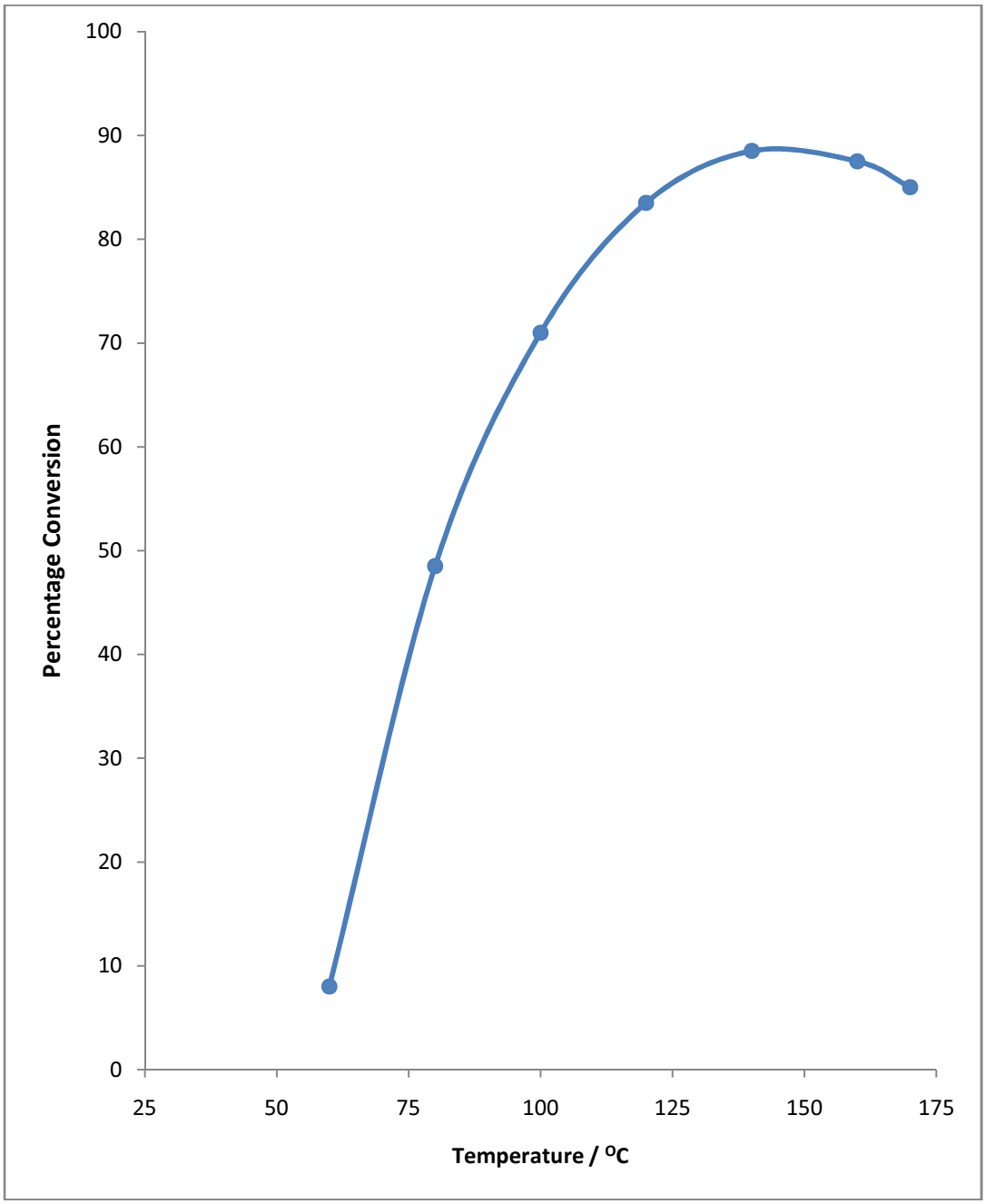
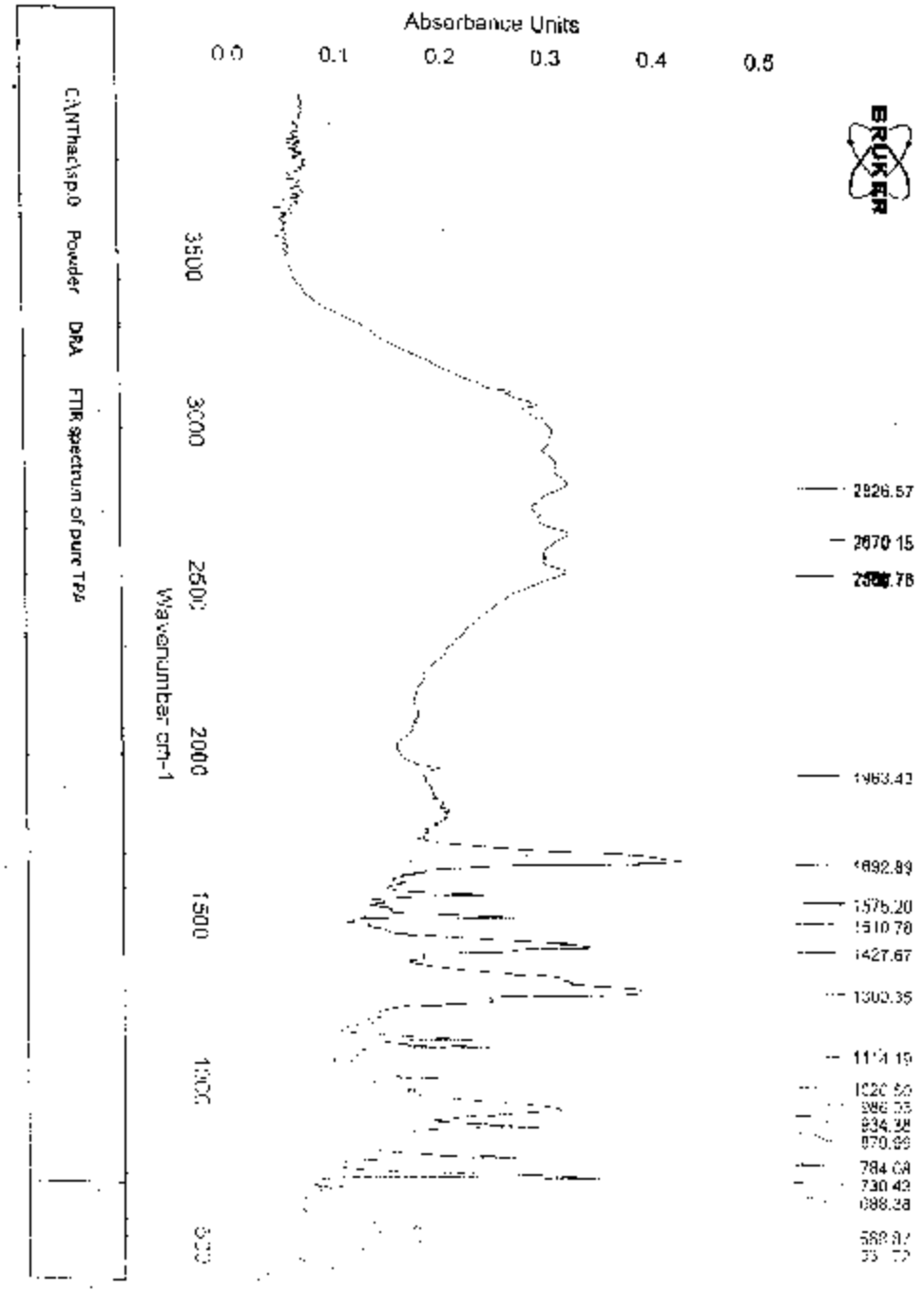


Fig. 5: Conversion of PET waste powder to terepthalic acid: Variation of temperature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0.2
0.3
0.4
0.5
BRUKER
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3500
3000
2500
2000
1500
Wavenumber cm-1
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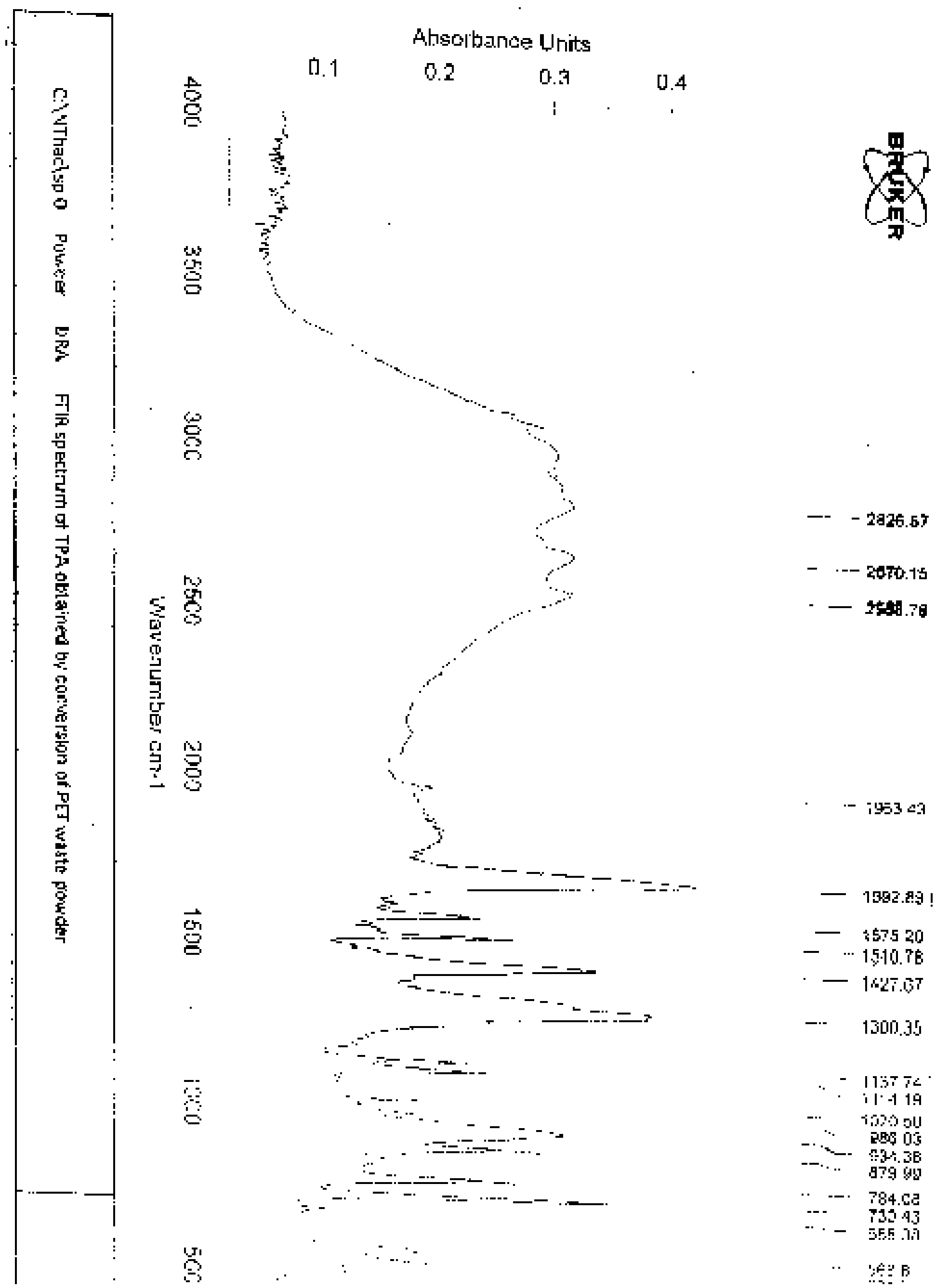
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1592.83
1575.20
1510.76
1427.67
1300.35
934.38
879.99
784.03
733.43
C:\NThac\sp 0 Powder DRA FTIR spectrum of TPA obtained by conversion of PET waste powder

RESULT AND DISCUSSION

Average molecular weight of Pet waste powder was determined by viscocity method. The plot of $\eta_{sp/c}$ versus percentage concentration of PET waste powder in phenol and tetrachloroethane (v/v) is plotted. The intercept on y-axis of the plot gives the intrinsic viscocity and from which average molecular weight of PET waste powder is determined as below.

$[\eta] = KM^{\alpha}$

$$M^{\alpha} = \frac{[\eta]}{K}$$

$$\alpha \log M = \log [\eta] - \log K$$

$$\log M = \frac{\log [\eta] - \log K}{\alpha}$$

Molecular weight, $M = Antilog \frac{\log[\eta] - \log K}{\alpha}$

$= Antilog \frac{[17.5] - \log 23 \times 10^3}{0.73}$

$= 8851$

The average molecular weight of used PET waste bottle powder is 8851.
[Ref. Fig. 1]

The results obtained in Table 1 and Fig. 2 shows that the maximum conversion of PET waste powder to terepthalic acid occurred at 7.0g of sodium hydroxide. The yield obtained is 87.0%. Further increase in amount of sodium hydroxide, the % conversion of PET waster powder to terepthalic acid decreases. And found to be 75% at 9.0g of sodium hydroxide. Hence the maximum conversion obtained at 7.0g of sodium hydroxide.

The optimised amount of pyridine has been found to be 3.0 cm^3. Which maintains the pH of the reaction system to 14.

Variation of particle size of PET waste powder from 100 μm to 800 μm shows that decrease in particle size from 800 μm to 100 μm, there is decrease in percentage conversion of PET waste powder. From this it can be concluded that smallest size of the PET waste powder is preferable for the conversion. This is because the small size of the waste powder provides the maximum surface area, higher heat and mass transfer. (Ref. Table 2 and Fig. 3).

The increase in the reflux time increases the conversion of PET waste powder into teripthalic acid. The maximum conversion take place at 120 minutes. Simultaneously and partially oxidizes to oxalic acid as follows;
[Ref. Table 3 & Fig. 4]

It is also observed that increase in temperature increase the conversion of PET waste powder into terepthalic acid. The optimum temperature for the conversion is found to be 140°c. (Ref. Table 4 and Fig. 5).
The terepthalic acid obtained after conversion of PET waste was characterised by taking its melting point. The melting point was observed to be 299°c. It was also characterised by taking FTIR spectra. It is found that the spectra of terepthalic acid after conversion is very much similar to spectra of the authentic terepthalic acid.

CONCLUSION

A. The molecular weight of PET waste powder using phenol and tetrachloroethane as a solvent was found to be 8851.
B. The percentage conversion of PET waste powder into terepthalic acid is maximum at 7g of sodium hydroxide.
C. The optimised amount of pyridine for conversion is found to be 3.0 cm^3.
D. The minimum particle size of 100 μm of PET waste powder is favourable for the conversion.
E. The optimised reaction time for the conversion is found to be 120 minutes.
F. The maximum temperature for the conversion is 140^{o}c.

REFERENCES

1. Jadhav, J. Y.; Kantor S. W.; polyester, Thermopolymers, Encyclopaedia of polymer science and Engg., wiley Interscience, New York, 12, 217 (1986).
2. Detye, K. V., Raje H. M., Sharma M. D.; *Resour, Conserv*., 11, 136, (1984).
3. Alter, H.; Disposal and reuse of polymers, Encyclopaedia of Polymer science and Engg.; wiley Interscience, New York, 5, 103 (1986).
4. Alter, H.; Ingle, G.; and Kaiser, E. R.; solid waste management (UK), 64, (12) 706 (1974).
5. Nir, M. M.; Miltz, J.; and Ram, A.; Update on plastics and environment progress and trends, *Plast. Engg*., 49, 75 (1993).
6. Fujita, A.; Sato, M.; and Morakami, M.; Depolymerization of polyesters scrap, *Japan Patent*, 4,284,646 (1985).
7. Fujita, A.; Sato, M.; and Morakami M; process for Depolymerization of polyester scraps, *US. Patent*, 4,609, 680 (1986).
8. Fussler C. R.; Polymer and Environment, PRI conference on Plastic Recycling, London (1989).
9. Leaversuch R. D.; Modern plastic; 68, 40-43 (1991).